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Phenom Desktop SEM for Gunshot Residue and Cathodoluminescence Imaging and Analysis

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Gunshot residue (GSR) particles are the residue material from the discharge of a firearm, that is, solid microscopic particles ejected from the openings, gaps and clefts of firearms [1, 2]. GSR can arise from the explosive propellant, cartridge primer, metals found in the projectile, projectile jacket; cartridge casing and gun material (eg. gun barrel).

GSR particles are identified by two features, morphology and chemical composition. The presence of Pb, Sb and Ba in a particle is considered characteristic of firearms origin. The most common technique utilised for the detection and identification of GSR particles is scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS).

More recently, there have been environmental concerns over the use of lead in ammunitions, which consequently have led to the development of heavy metal free (HMF) ammunition. Adding further to the discussion, particles from metal-free primers may be indistinguishable from environmental particles. The changing of the ammunition is also inadvertently altering the final elemental composition of GSR with limited information available to identify lead-free and/or organic GSR and differentiate it from environmental particles. In order to re-evaluate particle morphology and to distinguish GSR from environmental particles, detailed morphological and chemical characterization of these lead-free GSR particles and environmental particles is needed if the source of an unknown particle is to be determined with confidence.

Heavy metal free (HMF) particles use a Sintox primer which is specified as a non-toxic composite. This type of ammunition is based on tetrazene as a sensitizer, diazodinitrophenol (DDNP) instead of lead tricinate as an initiating explosive, and a pyro system consisting of zinc peroxide with titanium instead of lead styphnate, barium nitrate and antimony sulphide [6].

Sintox has a combination of tetrazene ($C_2H_6N_{10}.H_2O$), diazodinitrophenol ($C_6H_2N_4O_5$), zinc peroxide (ZnO_2 and ZnO, $Zn(OH)_2$) and 40 μm Ti particles. These result in GSR reaction products of ZnO, $ZnTiO_3$, $Zn_2Ti_3O_8$, TiO_2, $TiZn_2O_4$, as well as a nitrogen containing glass [1, 3, 4]. The problem with this reaction is the GSR products produced are very similar to particles from environmental sources. Consequently, researchers are looking into phase analysis through use of electron backscattered diffraction (EBSD) and also EDS [4-7].

Over the last couple years, there have been some significant improvements in desktop SEM design and capabilities with the addition of microanalysis systems. More recently, an automated gunshot residue system (GSR) system has been constructed and interfaced to the Phenom SEM (with automated stage) and a silicon drift detector (SDD). The system is also being used for particle searching in other applications. The Phenom XL is equipped with a CeB_6 source that enables extremely stable operation. This type of source has a typical operational life time of >1500 hours, which is ideal from a usability,

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serviceability and up-time perspective, and aids in detection of smaller GSR particles due to the brighter source (x10 compared to a W filament) and higher resolution [1-2].

The combination of a brighter source, the use of high-count rate, large-area, high-resolution SDDs, allows X-ray collection times to decrease to less than 1 second for GSR particles. In addition, the use of electrostatic scan with no hysteresis and the use of only the microscope’s scan generator communicating with the EDS, XRM and GSR system, has now produced a GSR analysis system that is capable of analysing very rapidly with a 30-50% decrease in analysis time [1-3].

With the advent of heavy metal free (HMF) ammunition and the investigation of organic residues, it is becoming more difficult to use basic SEM /EDS and GSR methods alone. Other techniques are required such as cathodoluminscence (CL) and Raman for the analysis of some of these types of ammunition [1, 3]. The SEM and optical systems, or the BSD electron signal, allow the user to find particles of interest, the EDS to determine their elements present, and the CL and/or Raman to distinguish between polymorphs and to validate non-detectable elements.

Recently, the SED detector on the Phenom instrument has been modified to detect CL by disconnecting the high tension (HT) bias power and removing the YAG scintillator and just leaving the light pipe that goes to the photomultiplier tube (PMT) (Figure 1c). This paper will discuss modifications that have been completed to obtain a CL detector and the subsequent implantation of a CL spectroscopy system. The preliminary results obtained from the CL detector from investigating GSR particles (Figure 2) will be presented.

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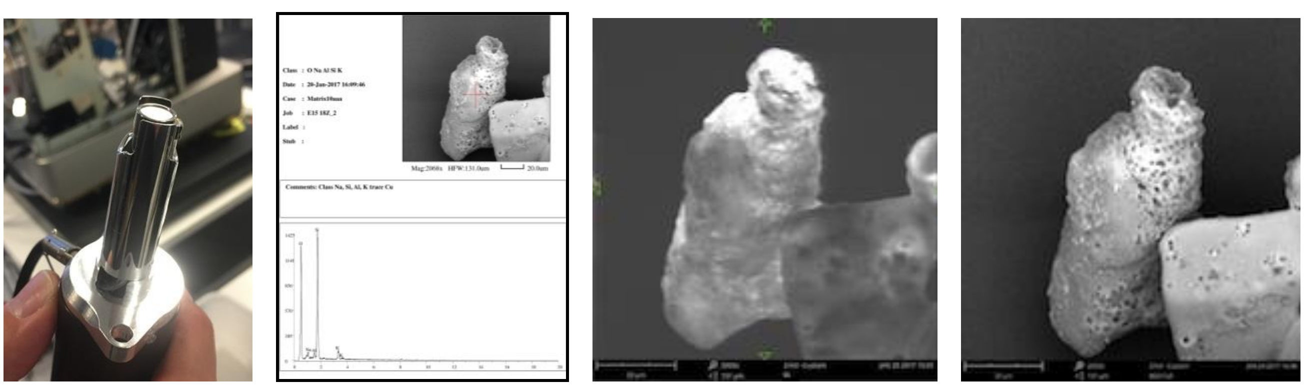

a. b. c. d.

Figure 1. a) SED detector before modification into a CL Detector, b) BSE image and EDS result of the particle using GSR software, c) CL image and d) BSE image.

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